

and FFA stability. We feel now that these difficulties have been overcome by using a very weak solution of soda ash as a stabilizer, which is injected with the water and oil at the base of the high-speed mixer. In most cases the refining loss is lower and color has a slight rise, but the bleach goes down in transit. It is not readily apparent why this weak solution of soda ash has been so effective. It improved not only

operation of the dryer but of all equipment in the degumming plant. The amount of soda ash used does not affect the free fatty acids of our crude oil and is used only as a stabilizing agent.

Any constructive criticisms or suggestions will be greatly appreciated.

[Received April 22, 1955]

## Meal Recycling—Method of Solvent-Extracting Oilseeds of High Fat Content: Application to Filtration-Extraction of Peanuts<sup>1</sup>

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**P**RESENT-DAY PRACTICE of solvent-extracting vegetable oilseeds requires many preparatory steps prior to extraction. Included in this category are cracking, flaking, moisture adjustment, cooking, drying, cooling, prepressing, and reforming (2, 3, 4, 7, 8). These steps are costly, and they involve special machinery, extra labor and power, especially when applied to high fat content oilseeds such as peanuts, sesame, castor, etc. Improper conditioning during preparation has adverse effects on the properties of unsaturated oil from seed such as flax and tung (9) and on protein which is readily susceptible to heat damage (1).

To simplify preparation there is need for a solvent-extraction process that eliminates costly machinery used in cooking or prepressing, reduces operating expenses, and efficiently extracts raw flakes or grit-like material. Such a process should be versatile enough to be applicable to many oilseeds without affecting the yields and properties of the oil and extracted meal products. These advantages are reported for a new process in which filtration rates are increased by the addition of solvent-extracted and dried oilseed meal.

In the filtration-extraction of raw flakes prepared from oilseeds of high oil content, *i.e.*, peanuts, the flakes consolidate, forming a solid mass, impervious to the flow of solvent. By mixing back with the unextracted raw flakes portions of the materials which have been solvent-extracted and completely desolventized by drying, the extraction rate is substantially increased and the effect of excessive fines is minimized. The coarseness and firmness of all extracted materials can be controlled in part by temperature and moisture control in the dryers. The use of such a process eliminates prepressing, cooking, resizing, and reforming. This method was applied to the filtration-extraction of raw peanuts, and the data show that adding extracted and dried material back to raw peanut flakes increased the mass velocity during filtration from an impractical 112 to a highly practical 2,860 lb. per square foot per hour (a 25-fold increase), yielding an extracted meal containing less than 1% residual lipids.

### Experimental Method and Equipment

Filtration-extraction tests were conducted, using the bench-scale test unit consisting of a metal funnel

with a removable filter (5). The raw peanut flakes were mixed with dried solvent extracted meal prior to slurring with commercial hexane.

**Materials.** The materials used were U. S. No. 1 shelled Spanish peanuts and solvent-extracted peanut meal obtained from a commercial solvent extraction plant. The Spanish peanuts were cracked and flaked to 0.010 in. in thickness. The solvent-extracted meal had been prepared from peanuts by a) cracking, b) heating mildly to about 140°F. with steam to a moisture of 7–8%, c) flaking, d) extracting with hexane, and e) heating the extracted flakes to about 200°F. This meal had an analysis of 8.89% water and 1.9% lipids and yielded a filtration mass velocity of 5,810 lb. per square foot per hour. Table I shows wet screen analyses of the peanut flakes containing 6.5% moisture and of the commercial solvent extracted meal.

TABLE I  
Wet Screen Analyses of Peanut Materials

Sample	Commercial Solvent-Extracted Meal, %	Raw Flakes, %
Screen Mesh		
5.....	2.2	2.5
8.....	5.0	6.1
16.....	26.2	26.5
20.....	22.2	10.6
40.....	24.8	13.5
60.....	11.7	11.3
80.....	2.2	4.0
120.....	1.8	4.5
170.....	1.1	4.6
200.....	0.2	2.0
300.....	0.5	1.0
Thru 300.....	2.1	13.5
Total.....	100.0	100.0

### Results

**Processing Variables in Filtration-Extraction.** Table II shows the effects of the following variables on the filtration-extraction of raw peanut flakes mixed with extracted peanut meals: a) types of extracted meal, b) amount of extracted and dried meal, and c) temperature of extraction. The addition of solvent-extracted meal (solvent-wet-extracted meal) did not materially increase the mass velocity over that obtained for raw flakes. Changing the ratio of raw peanut flakes (with 6.5% moisture) to solvent-extracted and dried peanut meal from 1/0 to 4/3 increased the mass velocity during filtration-extraction at 82°F. from 112 to 2,860 lb. per square foot per hour (a 25-fold increase). Extraction of the 4/3 ratio

<sup>1</sup> Presented at 29th annual fall meeting, American Oil Chemists' Society, Philadelphia, Oct. 10–12, 1955.

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TABLE II  
 Bench-Scale Filtration Extraction of Peanuts<sup>a</sup>

Test No.	1	2	3	4	5	6	7	8	9	10	11
Raw flakes, H <sub>2</sub> O, %.....	6.5	6.5	6.5	6.5	6.5	6.5	6.5	6.5	6.5	8.8	8.8
Wt., g. ....	450	337	250	200	200	200	200	200	200	450	337
Wt. extracted meats, g. ....	0	62.5	113	150	150	150 <sup>b</sup>	150	150 <sup>c</sup>	150 <sup>d</sup>	0	62.5
Ratio of raw to extracted meats.....	.....	5.4:1	2:1	4:3	4:3	4:3	4:3	4:3	4:3	.....	5.4:1
Filtrating time, thru 3 washes + 10 sec. ....	594 <sup>e</sup>	400	150	61	60	417	51	83	53	68	43
Extraction temperature, °F. ....	80	80	80	82	145	80	80	80	80	80	140
Cake thickness, in. ....	2%	2½	2	2½	2½	2%	2	2	2%	2%	2½
Screen mesh, size.....	24 x 110	24 x 110	24 x 110	24 x 110	24 x 110	24 x 110	60 x 60	60 x 60	60 x 60	60 x 60	60 x 60
Solvent to feed ratio.....	1.5:1	1.7:1	1.86:1	1.93:1	1.93:1	1.93:1	1.93:1	1.93:1	1.93:1	1.5:1	1.7:1
Mass velocity, lbs./ft. <sup>2</sup> /hr. ....	112	241	1162	2860	2840	419	3439	2095	3239	2580	4130
Extracted cake											
H <sub>2</sub> O, %.....	.....	8.5	.....	8.5	8.4	8.3	7.2	7.6	7.7	5.8	6.7
Lipids, %.....	.....	1.06	.....	1.35	0.80	0.72	1.37	0.98	1.74	4.4	2.46

<sup>a</sup> In all tests the weight of hexane used for slurring and each of the 3 washes was 675 g., the slurring time was 30 minutes and a vacuum of 4 in Hg was applied.

<sup>b</sup> Extracted marc from Test No. 1 (300 g. as is or 150 g. on a dry weight basis).

<sup>c</sup> 20- to 80-mesh portion of extracted and dried material.

<sup>d</sup> Coarser than 20- to 80-mesh portion of extracted and dried material.

<sup>e</sup> Extraction stopped, filtration too slow.

mixture at a temperature of 145°F. gave a mass velocity of 2,840 lb. per square foot per hour with residual lipids of 0.80% as compared to 1.35% for the extraction at 82°F. The added material was separated into coarse and fine fractions. Rates of filtration with the coarse fraction were higher than those obtained with its fine fraction. On raw peanut flakes moistened to 8.8% a change in the ratio of raw peanut flakes to solvent-extracted and dried peanut meal from 1/0 to 5.4/1 increased the rate of filtration from 2,580 to 4,130 lb. per square foot per hour (an increase of one-and-one-half times) and lowered the residual lipids in the extracted cake from 4.4 to 2.46%.

### Discussion

These experiments show that flake meats from raw peanuts can be processed by filtration-extraction if extracted and dried peanut meal are added to the unfatted peanut flakes. This method is simple, and the filtration rates obtained are within practical limits. In the preparation of the commercial extracted and dried peanut meal, indications are that the mild heating treatment may be eliminated.

These data indicate that raw peanuts can be extracted without prepressing, or cooking and reforming, thus indicating elimination of possible expensive preparation equipment, such as cookers or expellers and reformers. The coarseness of the extracted meal added is an important factor and is partially a result of temperature and moisture control of the desolventizer and final moisture content of the dried meal. The addition of moisture to the oil-free marc prior to entering the dryer will reduce the amount of fines formed in the final product. With this recycling method of extraction in which high temperatures are avoided, a high-grade, solvent-extracted peanut meal can be obtained which should be suitable for industrial or food utilization. High moisture raw peanut flakes have less "fines," are very pliable and easily compressible and are somewhat difficult to handle mechanically without packing. They give greater filtration rates than those with low moisture, but the extracted meals resulting have high residual lipids.

Even with a high proportion of extracted and dried peanuts mixed back into the incoming feed (4/3 ratio of raw peanuts to extracted peanuts) and the increased load on the dryers, calculations indicate a 100-ton a day cottonseed or 50-ton a day soybean filtration-extraction plant should handle 45 tons a day of peanuts. The continuous horizontal filter may have to be slightly larger than that required for cot-

tseed. The final oil miscella concentrations would have a lower oil content than would be expected from conventional solvent extraction, which would mean that additional solvent would have to be evaporated and thus require more steam. Any reduction in the amounts of material added back to the feed would increase the amount of peanuts that could be extracted.

This method of adding back solvent-extracted and dried peanuts should be applicable to any type of oilseed and particularly to those containing drying oils. Drying oils under some conditions tend to oxidize or polymerize to some extent in cooking and drying of rolled oilseed meats. Any polymerized or oxidized oil formed is not readily extractable (9), thus the extraction efficiency may be reduced. The adding back method while applied to filtration-extraction should aid any type of percolation extraction.

### Summary and Conclusions

Raw peanuts may be processed by filtration-extraction on a bench scale if solvent-extracted and dried peanut meal is added to the raw flakes prior to extraction. Prepressing, cooking, resizing, reforming, etc., prior to extraction are eliminated. The method while applied to peanuts should be applicable to any type of oilseed and makes possible the filtration-extraction of raw oilseed meats previously considered impossible. Disadvantages are that there would be an increased load on meal dryers, and additional solvent would have to be evaporated due to low oil content of the miscella. The data show that increasing the moisture content of peanut meats decreases the amount of extracted and dried material to be added back to the incoming raw meats but increases the residual lipids in the extracted meals. Indications are that the moisture content of raw peanuts might be adjusted to reduce the amount of solvent-extracted meal needed for recycling and yet yield an extracted meal with low residual lipids. The data show that practical mass velocities of approximately 3,000 lb. per square foot per hour with residual lipids of less than 1% can be obtained, using a solvent to meal ratio of 1.93 to 1 on a 4/3 ratio mixture of raw peanut flakes (6.5% water) to extracted meal. Calculations from the laboratory data indicate that a 100-ton a day cottonseed filtration-extraction plant should process approximately 45 tons of peanuts.

### Acknowledgment

The authors wish to express their appreciation to A. V. Graci Jr. for his assistance in performing the

filtration-extraction tests and to Marco L. Paredes for the analyses reported.

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[Received June 13, 1955]

## ABSTRACTS

R. A. Reiners, Editor

### • Oils and Fats

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**For top-quality fried foods curb these six trouble factors.** W. A. Quammen (Res. and Dev. Dept., Procter and Gamble Co., Cincinnati, Ohio). *Food Eng.*, **27**, 76-77 (1955). The tendency of a bland stable frying fat to darken, foam, smoke, develop poorer flavor and produce gum on equipment is accelerated by (1) excessive heat, (2) metallic contamination, (3) soap and caustic residues left on equipment after cleaning operation, (4) burnt food particles, (5) inadequate filtering and (6) insufficient turnover. For most efficient operation complete turnover of fat in about 10 hours is required.

**Separation of triglycerides by countercurrent distribution.** J. J. Taber (University of Pittsburgh, Pittsburgh, Pa.). *Univ. Microfilms* (Ann Arbor, Michigan), Publ. No. 11609, 176 pp. *Dissertation Abstr.* **15**, 727-8 (1955). (*C. A.* **49**, 11,301)

**Preparation of tasty vegetable products by deep fat frying.** E. G. Kelley and R. R. Baum (Eastern Reg. Res. Lab., Philadelphia, Pa.). *Food Tech.* **9**, 388-92 (1955). Beet, carrot and parsnip slices have been cooked in deep fat to yield tasty and attractive vegetable chips. Peas and lima beans have been prepared as crunchy pieces having a nut-like flavor and appearance. When cooked in a stabilized oil, the fried vegetables are stable over 6-12 months at room temperature.

**Catalysts. X. Rate and selectivity of hydrogenation of sunflower oil in the presence of nickel formate catalyst.** Z. Csuros, I. Géczy and D. Szabó (Tech. Univ., Budapest). *Acta Chim. Acad. Sci. Hung.* **2**, 33-56 (1952). A catalyst consisting of 50% Ni in hydrogenated sunflower oil was prepared. The rate of hydrogenation of sunflower oil with 0.4% catalyst was increased by raising the temperature from 160° to 200° and declined above 200°. An induction period was present at 120-40°. By controlling the rate of hydrogenation, sunflower oil could be selectively reduced so that only one double bond in the linoleic acid became saturated. Selectivity increased with larger quantities of the catalyst up to 0.6% and with a rise in temperature up to 220°; above this temperature and concentration selectivity declined. (*C. A.* **49**, 11,300)

**Component fatty acids of the oil from the seeds of Benincasa cerifera.** S. C. Sethi and J. S. Aggarwal (National Chem. Lab. India, Poona). *J. Sci. Ind. Research* **13B**, 853-4 (1954). Extractions of the seed kernels with petroleum ether gives a yellow oil  $n_D^{20}$  1.4739,  $d_4^{20}$  0.9196, and unsaponifiable matter 1.47% (identified as stosterol; acetate, m. 130°). The fatty acid content of the yellow oil is linolenic 1.02, linoleic 62.36, oleic 20.03, palmitic 10.56, stearic 5.76, and arachidonic 0.27%. (*C. A.* **49**, 11,299)

**Studies on the oxidation mechanism in emulsified fat or oil. I. The influence of milk constituents on oxidation in margarine.** Y. Ozawa, H. Kembo and K. Naito. *Bull. Natl. Inst. Agr. Sci., Jap.*, Ser. G, **28**, 67-74 (1953). Of several milk constituents tested for antioxidant properties in margarine enriched with vitamin A, the most effective were skim milk, buttermilk, and sodium caseinate, and their action was equal to that of propyl gallate. The seat of this activity was the protein, probably the casein, and since the antioxidant properties were not affected by heating, no correlation with oxidation-reduction potential was indicated. (*C. A.* **49**, 11,199)

**II. The influence of milk and egg constituents on the catalytic oxidation under the existence of copper ions.** *Ibid.* 75-81. The

rate of oxidation increased when 1-4 p.p.m. of Cu were added to margarine. The fats were protected by colloidal protein solutions (e.g., egg white, egg yolk, sodium caseinate, skim milk, etc.), their antioxidant activity increasing with increased emulsifying powers. Egg yolk and sodium caseinate were the most effective. (*C. A.* **49**, 11,199)

**Antioxidants and their detection.** H. Janecke (Univ. Frankfurt/Main, Ger.). *Deut. Lebensm.-Rundschau* **51**, 121-4 (1955). The means for detecting the four most frequently used antioxidants, i.e. dihydronorguaiaretic acid, butoxyanisole, some gallates and mixtures of tocopherol are discussed. (*C. A.* **49**, 11,195)

**The detection and quantitative determination of coloring materials in butter and margarine.** H. M. Espoy and H. M. Barnett (Barnett Lab., Long Beach, California). *Food Tech.* **9**, 367-72 (1955). A method has been developed for the detection and quantitative estimation of the principal pigments found or used in commercial butters and margarines. Mixtures of carotene, annatto extract, and coal tar colors can be separated and analyzed individually without the necessity of saponifying and extracting the oil carrier. A number of samples of butter and margarine were analyzed by these methods for type and strength of coloring materials.

**Multicolumn continuous distillation.** R. Dobrowolski. *Przemysl Chem.* **9**, 419-26 (1953). Multicolumn apparatus was applied to distillation of crude fatty acids. Five small columns were used; in each one only one fraction was fractionated. The most corrosive acids ( $C_7-C_8$ ) were distilled in the first column made of porcelain, and the least corrosive acid,  $C_{22}$ , was distilled in the fifth column made of chrome steel. The advantages of this distillation are: (1) differentiation of column according to variables as temperature and vacuum, (2) differentiation of the material used in producing the columns, and thus saving of the acid-proof materials, (3) lesser use of thermal energy, (4) better quality of the products, and (5) saving of steel. (*C. A.* **49**, 10,671)

**The branched-chain fatty acids of ox fat. 2. The isolation of 15-methylhexadecanoic acid.** R. P. Hansen, F. B. Shorland, and N. June Cooke (Fats Research Lab., Dept. of Scientific and Ind. Res., Wellington, New Zealand). *Biochem. J.* **61**, 141-143 (1955). The  $C_{17}$  branched-chain fatty acid 15-methylhexadecanoic acid has been isolated in trace quantities (approximately 0.06% of total fatty acids) from hydrogenated ox perinephric fat.

**Determination of carboxylic acid anhydrides by reaction with morpholine.** J. B. Johnson and G. L. Funk (Chemical & Physical Methods Lab., Carbide & Carbon Chemicals Co., South Charleston, W. Va.). *Anal. Chem.* **27**, 1464-5 (1955). A titrimetric method employing an indicator was sought for the direct determination of carboxylic acid anhydrides in the presence of and to the exclusion of the corresponding acids. A satisfactory method was developed, based on the reaction of the anhydride with morpholine and titration of the excess reagent with methanolic hydrochloric acid. Data are presented on the determination of the purity of eight anhydrides and the determination of low concentrations of acetic anhydride in glacial acetic acid. The procedure is rapid, generally applicable to a variety of acid anhydrides, and accurate and precise over a wide range of concentrations.

**The component fatty acids of the fat of *Aspergillus nidulans*.** J. Singh, T. K. Walker, and M. L. Meara (College of Technology, Univ. of Manchester, England). *Biochem. J.* **61**, 85-88 (1955). The fat elaborated by *A. nidulans* grown on a sucrose medium is remarkably low in free acidity. The component acids